

Modelling and Simulation of ethyl acetate reactive distillation column using ASPEN PLUS

*A Project submitted to the
National Institute of Technology, Rourkela*

*In partial fulfilment of the requirements
of*
Bachelor of Technology (Chemical Engineering)

By

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CERTIFICATE

This is to certify that the thesis entitled **Modeling and Simulation of Ethyl Acetate Reactive Distillation Column Using ASPEN PLUS**, submitted by **Shyam Kumar** to National Institute of Technology, Rourkela is a record of bonafide project work under my supervision and is worthy for the partial fulfillment of the degree of Bachelor of Technology (Chemical Engineering) of the Institute. The candidate has fulfilled all prescribed requirements and the thesis, which is based on candidate's own work, has not been submitted elsewhere.

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ABSTRACT

In this thesis, we study the modeling and simulation of a reactive distillation column for the production of ethyl acetate from acetic acid and ethyl alcohol using ASPENPLUS. Starting from a conventional configuration, which involves feeding in a single tray, different configuration is proposed and various specifications are studied for the attainment of higher conversion and purity at the steady state. In ASPEN DYNAMICS an analysis of the column dynamics is then performed. Cascade control structure is studied for the base design.

CONTENTS

| | |
|--|------|
| Cover page | i |
| Certificate | ii |
| Acknowledgement | iii |
| Abstract | iv |
| Contents | v |
| List of figures | vi |
| List of tables' | vii |
| Nomenclature | viii |
| 1. Introduction and Literature | |
| 1.1 Background of Reactive Distillation | 2 |
| 1.2 Commercial applications of Reactive Distillation | 3 |
| 1.3 Advantages of Reactive Distillation | 4 |
| 1.4 Constraints and disadvantages of Reactive Distillation | 5 |
| 1.5 Properties and uses of ethyl acetate | 5 |
| 1.6 Production of ethyl acetate | 7 |
| 1.7 ASPEN PLUS software | 8 |
| 1.8 Aspen Dynamics | 9 |
| 2. Modeling | 10 |
| 3. Steady state simulation | |
| 3.1 Configuration-1 | 17 |
| 3.2 Configuration-2 | 23 |
| 3.3 Configuration-3 | 25 |
| 3.4 Configuration-4 | 28 |

| | |
|---|----|
| 4. Dynamic simulation | |
| 4.1 Installing level controller | 34 |
| 4.2 Installing flow controller | 35 |
| 4.3 Installing temperature controller | 35 |
| 4.4 Installing composition controller | 36 |
| 4.6 Cascade Control Structure | 36 |
| 4.7 Performance Evaluation of Control Structure | 37 |
| 5. Conclusion | 39 |
| References | 40 |

List of figures:

| Figure no. | Description | Page no. |
|------------|--|----------|
| 2.1 | Configuration for reactive-distillation column | 11 |
| 2.2 | General reactive-distillation stage | 12 |
| 3.1 | Single feed Flow diagram in aspen plus user interface for Reactive Distillation | 18 |
| 3.2 | Stage wise Composition profile | 18 |
| 3.3 | Stage wise temperature profile | 19 |
| 3.4 | Effect of reflux ratio on liquid composition of ethyl acetate | 19 |
| 3.5 | Effect of distillate flow rate on liquid composition of ethyl acetate in distillate and bottom | 21 |
| 3.6 | Effect of total no of stages in column on liquid composition of ethyl acetate in distillate and bottom | 22 |
| 3.7 | Composition profile of ethyl acetate in liquid phase for different activity model | 23 |
| 3.8 | Double feed Flow diagram in aspen plus user interface for | 24 |

| | | |
|------|--|----|
| | Reactive Distillation | |
| 3.9 | Double Reactive distillation column diagram in aspen plus user interface | 25 |
| 3.10 | Stage-wise liquid composition profile of ethyl acetate in RD-1 and RD-2 | 26 |
| 3.11 | Stage-wise liquid composition profile of ethyl acetate in RD-1 and RD-2 with different feed location | 27 |
| 3.12 | Reactive distillation column with heated side-stream in aspen plus user interface | 28 |
| 4.1 | Initial screen in Aspen Dynamics for single column with single feed | 33 |
| 4.2 | Configuration-1 with cascade control structure in ASPEN DYNAMICS | 37 |
| 4.3 | Dynamic response of composition in bottom and distillate subjected to feed flow disturbance | 37 |
| 4.4 | Dynamic response of composition in bottom and distillate subjected to feed composition disturbance | 38 |
| 4.5 | Dynamic response of composition in bottom and distillate subjected to distillate set-point disturbance | 38 |

List of tables:

| | |
|---|----|
| Table 1(simulation result for changing distillate flow rate on composition) | 20 |
| Table 2(simulation result for changing total no of stages in column on composition) | 21 |
| Table 3(simulation results for different activity model on composition) | 22 |
| Table 4 (simulation result for double feed reactive distillation column) | 25 |
| Table-5(study result of varying ΔT across Heat Exchanger on composition) | 30 |
| Table 6,(a,b)(study result of pumparound return location on composition) | 31 |

Nomenclature:

A_p = The Arrhenius pre-exponential factor for the p th term

E_p = Energy of activation for the p th term

E_i = The discrepancy (residual) of the energy equation for stage i

$f_{i,j}$ = Liquid component molar flow for component j in the fresh feed to stage i

$$F_i = \sum_{j=1}^c f_{i,j}$$

$g_{i,j}$ = Vapor component molar flow for component j in the fresh feed to stage i

$$G_i = \sum_{j=1}^c g_{i,j}$$

H_i = Enthalpy of the primary vapor leaving stage i ; i.e. not including the vapor sidestream

H_{G_i} = Enthalpy of G_i

h_i = Enthalpy of the primary liquid leaving stage i ; i.e. not including the sidestream or entrained liquid

h_{F_i} = Liquid enthalpy of F_i ,

$K_{i,j}$ = Vapor-liquid equilibrium ratio for component j on stage i

$l_{i,j}$ = Moles of component j in the primary liquid leaving stage i ; i.e. not including sidestream liquid or entrained liquid

$$L_i = \sum_{j=1}^c l_{i,j}$$

$M_{i,j}$ = Discrepancy (residual) of the component material balance for component j and stage i

N = Number of stages including condensers and reboilers

NRX = Number of chemical reactions

P_i = Pressure on stage i

Q_i = Heat duty at stage i

S_i = Ratio of vapor drawoff to primary vapor (vapor not withdrawn or entrained) on stage i
 $= W_i / V_i$

s_i = Ratio of liquid drawoff to primary liquid (liquid not withdrawn or entrained) on stage i
 $= P_i / L_i$

T_i = Temperature on stage i

U_i = Liquid holdup on stage i

$v_{i,j}$ = Moles of component j in the primary vapor leaving stage i ; i.e. not including sidestream vapor

$$V_i = \sum_{j=1}^c v_{i,j}$$

$v_{i,j}$ = Stoichiometric coefficient for component j in chemical reaction n

$r_{i,n}$ = reaction rate for reaction n on stage j, as the increase in moles of a reference reactant per unit time per unit volume of liquid phase

x_j = Mol-fraction in liquid of component j

y_j = Mol-fraction in vapor of component j

$x_{i,j}$ = Mol-fraction in liquid of component j at stage i

$y_{i,j}$ = Mol-fraction in vapor of component j at stage i

CHAPTER-1

INTRODUCTION AND LITERATURE

Chapter-1

Introduction and literature

Reactive distillation involves simultaneous chemical reaction and distillation. The chemical reaction usually takes place in the liquid phase or at the surface of a solid catalyst in contact with the liquid phase ^[1]. General application of reactive distillation is the separation of a close-boiling or azeotropic mixture described by Terril *et al.* ^[2]. A second application of reactive distillation involves taking into account undesirable reaction that may occur during distillation but the most interesting application involves combining chemical reactions and separation by distillation in a single distillation apparatus ^[1]

The technique offers a key opportunity for improving the structure of a process. It is a so-called hybrid process, i.e. it merges two different unit operations in a single apparatus, namely reaction and distillation. But the combination of distillation and reactions is possible only if the conditions of both unit operations can be combined.

Reactive distillation can be used with a wide variety of chemistries, including the following: Acetylation, Aldol condensation, Alkylation, Amination, Dehydration, Esterification, Etherification, Hydrolysis, Isomerization, Oligomerization, Transesterification.

1.1 Background of Reactive Distillation:

Reactive distillation, combination of chemical reaction and multicomponent distillation in a single unit, has proven to be advantageous over conventional process systems consisting of separate reactor and distillation unit. Prior to the advent of digital computers, the literature dealt mainly with proposed applications and simplified calculational procedures. This concept appears to have been first pronounced by Backhaus, who, starting in 1921, obtained a series of patents for esterification reaction in a distillation column. This concept of continuous and simultaneous chemical reaction and distillation in a single vessel was verified experimentally by Leyes and Othmer, for the esterification acetic acid with an excess of *n*-butanol in the presence of sulfuric acid catalyst to produce butyl acetate and water. ^[1]

Further research, both of an experimental and theoretical nature, was conducted during later years. Examples are hydrolysis of acetic anhydride-parasitic reaction during distillation(Marek^[8]),

esterification of acetic acid with ethanol (Suzuki *et al.*,^[9] Komatsu,^[10] Alejski *et al.*,^[11] Simandl and Svrcek,^[12]) and methanol (Corrigan and Ferris,^[13] Agreda *et al.*,^[14]), trans-esterification of butyl acetate and ethyl alcohol (Davies and Jeffreys,^[15]), synthesis of propylene oxide from propylene chlorohydrins (Carra *et al.*,^[16]), synthesis of epichlorohydrin from chlorohydrins (Carra *et al.*,^[17]) and Nylon 6,6 production (Grosser *et al.*,^[18]). The combination of reaction and distillation over a catalyst bed has been extensively investigated (Westerterp,^[19]). The most typical examples are MTBE and cumene production (Smith and Huddleston,^[20] DeGarmo *et al.*,^[22] Shoemaker,^[21]).

Modeling of reactive distillation has received considerable attention over the last 15 years and several key contributions have appeared in the literature (Doherty and Malone^[24] and the excellent overview of Noeres *et al.*,). Pilavachi *et al.* presented an extensive discussion of several important aspects that affect the accurate modeling of reactive distillation processes. Schenk *et al.* described in considerable details a hybrid-modeling environment in which a reactive distillation process can be simulated using a combination of equilibrium and mass transfer models, both in steady state and dynamic modes. Recently, Almeida-Rivera *et al.* presented a critical review of current available methodologies for designing reactive distillation processes.

The control of reactive distillation has received some attention in recent years. Al-Arfaj and Luyben^[23], Sneesby *et al.*,^[25] Kumar and Daoutidis,^[26] discussed the decentralized PI control structures for reactive distillation column. Al-Arfaj and Luyben^[23] discussed the possibility of multiple steady states in many reactive distillation systems. The presence of multiplicities and the highly nonlinear nature of reactive distillation may impose limitations on use of linear controllers. Kumar and Daoutidis^[26] have discussed the superior performance of nonlinear controller compared to linear controller for reactive distillation systems.

1.2 Commercial applications of Reactive Distillation include following^[1]

1. The esterification of acetic acid with ethanol to produce Ethyl acetate and water.
2. The reaction of formaldehyde and methanol to produce methylal and water, using a solid acid catalyst, as described by Masamoto and Matasuzaki.
3. The esterification of acetic acid with methanol to produce methyl acetate and water, using sulfuric acid as catalyst, as patented by Agreda and Partin, and described by Agreda, Partin and Heise.

4. The reaction of isobutene with methanol to produce methyl-*tert*-butyl ether (MTBE), using a solid, strong-acid ion-exchange resin catalyst, as patented by Smith and further developed by DeGarmo, Parulekar, and Pinjala.

There are many documented success stories involving the industrial implementation of reactive distillation. The applications of reactive distillation in the chemical and petroleum industries have increased rapidly in the past decade. One such example is the manufacturing of methyl acetate by the Eastman Chemical Company. In this case a single reactive distillation column replaced the traditional flow sheet consisting of eleven major unit operations along with an assortment of heat exchangers, pumps and controllers. The result was a five-fold reduction in capital investment and energy consumption over the conventional design for methyl acetate production.

1.3 Advantages of Reactive Distillation:

1. Increased speed and improved efficiency.
2. Lower costs – reduced equipment use, energy use and handling.
3. Less waste and fewer byproducts
4. Improved product quality—reducing opportunity for degradation because of less heat; heat duty can be reduced by utilizing the heat of reaction (if present) in situ.
5. Recycle costs for excess reactant, which is necessary for a conventional reactor to prevent side reactions and chemical equilibrium limitation, can be reduced.
6. Reaction conversions can be increased by overcoming chemical equilibrium limitation through the removal of reaction products.
7. Limitation of azeotropic mixture can be overcome by reaction, described by Terril *et al.*^[1]

1.4 Constraints and disadvantages of Reactive Distillation:

In spite of above stated benefits of reactive distillation, cannot be used for every process that requires reaction and separation in a single unit. It has some constraints. In general, reactive distillation is not attractive for supercritical condition, for gas-phase reaction, and for reaction that must take place at high temperature and pressures, and/or that involves solid reactants or products^[1].

This type of reactive distillation is considered as an alternative to the use of separate reactor and distillation vessel whenever the following holds:

1. Feasible temperature and pressure for the reaction and distillation are the same. That is, reaction rates and distillation rates are of same magnitude. the reactions have to show reasonable data for conversions at pressure and temperature levels that are compatible with distillation conditions.
2. The chemical reaction occurs in the liquid phase, in the presence or absence of a homogeneous catalyst, or at the interface of a liquid and a solid catalyst.
3. The reaction is equilibrium-limited such that if one or more of the products formed can be removed, the reaction can be driven to completion; thus, a large excess of a reactant is not necessary to achieve a high conversion. This is particularly advantageous when recovery of the excess reagent is difficult because of azeotrope formation.
4. Higher requirements on the quality of the design and control systems including more sophisticated controller designs and more complicated control structures.
5. Residence time requirements but limited hold-up in distillation column.
6. Volatility constraints for reagents and products in the reaction zone of distillation column.

1.5 Ethyl acetate:

Ethyl acetate is the organic compound with the formula $\text{CH}_3\text{COOC}_2\text{H}_5$. This colorless liquid has a characteristic, pungent smell like certain glues or nail polish removers, in which it is used. Ethyl acetate is the ester from ethanol and acetic acid; it is manufactured on a large scale for use as a solvent. Ethyl acetate is a moderately polar solvent that has the advantages of being volatile, relatively non-toxic, and non-hygroscopic. It is a weak hydrogen bond acceptor, and is not a donor due to the lack of an acidic proton. Ethyl acetate can dissolve up to 3% water and has a solubility of 8% in water at room temperature. It is unstable in the presence of strong aqueous bases and acids. It is Soluble in most organic solvents, such as alcohol, acetone, ether and chloroform.

Properties and uses:

| | |
|-------------------|--------------------------------------|
| Molecular formula | $\text{CH}_3\text{COOC}_2\text{H}_5$ |
| Molar mass | 88.105 g/mol |

| | |
|---------------|-------------------------|
| Density | 0.897 g/cm ³ |
| Melting point | -83.6 °C |
| Boiling point | 77 °C |
| Viscosity | 0.426 cp at 25°C |

Ethyl acetate has a wide range of applications, across many industries, including:

Surface coating and thinners: Ethyl acetate is one of the most popular solvents and finds wide use in the manufacture of nitrocellulose lacquers, varnishes and thinners, to dissolve the pigments for nail varnishes. It exhibits high dilution ratios with both aromatic and aliphatic diluents and is the least toxic of industrial organic solvents.

Pharmaceuticals: Ethyl acetate is an important component in extractants for the concentration and purification of antibiotics. It is also used as an intermediate in the manufacture of various drugs.

Flavors and essences: Ethyl acetate finds extensive use in the preparation of synthetic fruit essences, flavors and perfumes.

Flexible packaging: Substantial quantities of ethyl acetate are used in the manufacture of flexible packaging and in the manufacture of polyester films and BOPP films. It is also used in the treatment of aluminum foils.

Occurrence in wines: Ethyl acetate is the most common ester found in wine, being the production of the most common volatile organic acid-acetic acid and the ethanol alcohol created during the fermentation of wine. The aroma of ethyl acetate is most vivid in younger wines and contributes towards the general perception of "fruitiness" in the wine. Sensitivity varies with most people having a perception threshold around 120 mg/lit. An excessive amount of ethyl acetate is considered a wine fault. Exposure to oxygen can exacerbate the fault due to the oxidation of ethanol creating acetaldehyde. This can leave the wine with sharp vinegar like taste.

Miscellaneous: Ethyl acetate is used in the manufacture of adhesives, cleaning fluids, inks, nail polish removers and silk, coated papers, explosives, artificial leather, photographic films & plates. In the field of entomology, ethyl acetate is an effective poison for use in insect collecting and study. In a killing jar charged with ethyl acetate, the vapors will kill the collected (usually adult) insect quickly without destroying it. Because it is not hygroscopic, ethyl acetate also keeps the insect soft enough to allow proper mounting suitable for a collection.

1.6 Production of ethyl acetate:

Ethyl acetate is produced by the esterification reaction of ethyl alcohol and acetic acid using catalysts such as sulphuric acid, Para toluene sulphonic acid or ion exchange resins. The reaction of ethanol (EtOH) with acetic acid (AcOH) towards ethyl acetate (EtAc) and water (H₂O) is an equilibrium reaction.



The system is strongly non-ideal due to the presence of ethanol, acetic acid, and water. The separation of pure components is very difficult due to the existence of five normal azeotropes, namely, ethanol–water; water–acetic acid, ethyl acetate–ethanol, ethyl acetate–water, and ethanol–ethyl acetate–water. Sujuki *et al.* ^[9] also determined the phase equilibrium for the system taking the reaction into account (they fitted 16 coefficients in Modified Margules equations, for calculating the VLE-model of this mixture).

The normal boiling point of ethyl acetate, ethanol, water and acetic acid is 77.1, 78.4, 100 and 118.1 in °C respectively.

The order of volatility is ethyl acetate, ethanol, water and acetic acid. Ethanol and water do not differ greatly in volatility, making it difficult to move ethanol up the column.

The minimum-boiling binary homogeneous azeotropes are formed by ethanol–water at 78.2°C With 10.57mol% water and by ethyl acetate–ethanol at 71.8°C with 46 mol% ethanol. A minimum-boiling binary heterogeneous azeotrope is formed by ethyl acetate–water at 70.4°C with 24mol% water, and a ternary, minimum-boiling azeotrope is formed by ethanol–ethyl acetate–water at 70.3°C with 12.4mol% ethanol and 60.1 mol% ethyl acetate. ^[1]

The main problems encountered in achieving high purity products in the ethyl acetate reactive distillation system are summarized subsequently (Bock *et al.* 1997, Chang and Seader ^[29]):

- Unfavorable reactant conversion;
- Similar *K*-values of ethanol, water, and ethyl acetate;
- Temperature profile in the column;

- The system is strongly non-ideal

1.7 ASPEN PLUS software:

In 1970s the researchers at MIT's Energy Laboratory developed a prototype for process simulation. They called it Advanced System for Process Engineering (ASPEN). This software has been commercialized in 1980's by the foundation of a company named AspenTech. ASPEN PLUS offers a complete integrated solution to chemical process industries. This sophisticated software package can be used in almost every aspect of process engineering from design stage to cost and profitability analysis. It has a built- in model library for distillation columns, separators, heat exchangers, reactors, etc. Custom or propriety models can extend its model library. These user models are created with FORTRAN subroutines or Excel worksheets and added to its model library. Using Visual Basic to add input forms for the user models makes them indistinguishable from the built- in ones. It has a built- in property databank for thermodynamic and physical parameters. During the calculation of the flow sheet any missing parameter can be estimated automatically by various group contribution methods.

Aspen Plus can interactively change specifications such as, flow sheet configuration, operating conditions, and feed compositions, to run new cases and analyze process alternatives Aspen Plus allows us to perform a wide range of tasks such as estimating and regressing physical properties, generating custom graphical and tabular output results, fitting plant data to simulation models, optimizing process, and interfacing results to spreadsheets.

1.8 Aspen Dynamics:

Aspen Dynamics complements the steady-state simulation capabilities of Aspen Plus and delivers the benefits of dynamic modeling to the Petrochemicals, Chemicals, and Specialty Chemicals industries throughout plant operation and engineering organizations.

In Aspen Dynamics we can easily:

- Add, change and remove control elements
- Select measured and manipulated variables from selection lists
- Configure cascade control loops
- Import control structures from other Aspen Dynamics generated input files

Aspen Dynamics is a state-of-the-art solution designed specifically for dynamic process simulation. Aspen Dynamics is tightly integrated with Aspen Plus, Aspen Tech's steady-state simulator for the chemical process industries. This integration enables users to use an existing Aspen Plus steady-state simulation and quickly create a dynamic simulation. This enables users to fully leverage their existing investments in steady-state Aspen Plus models and ensures consistency with their steady-state simulation results.

CHAPTER-2

MODELING

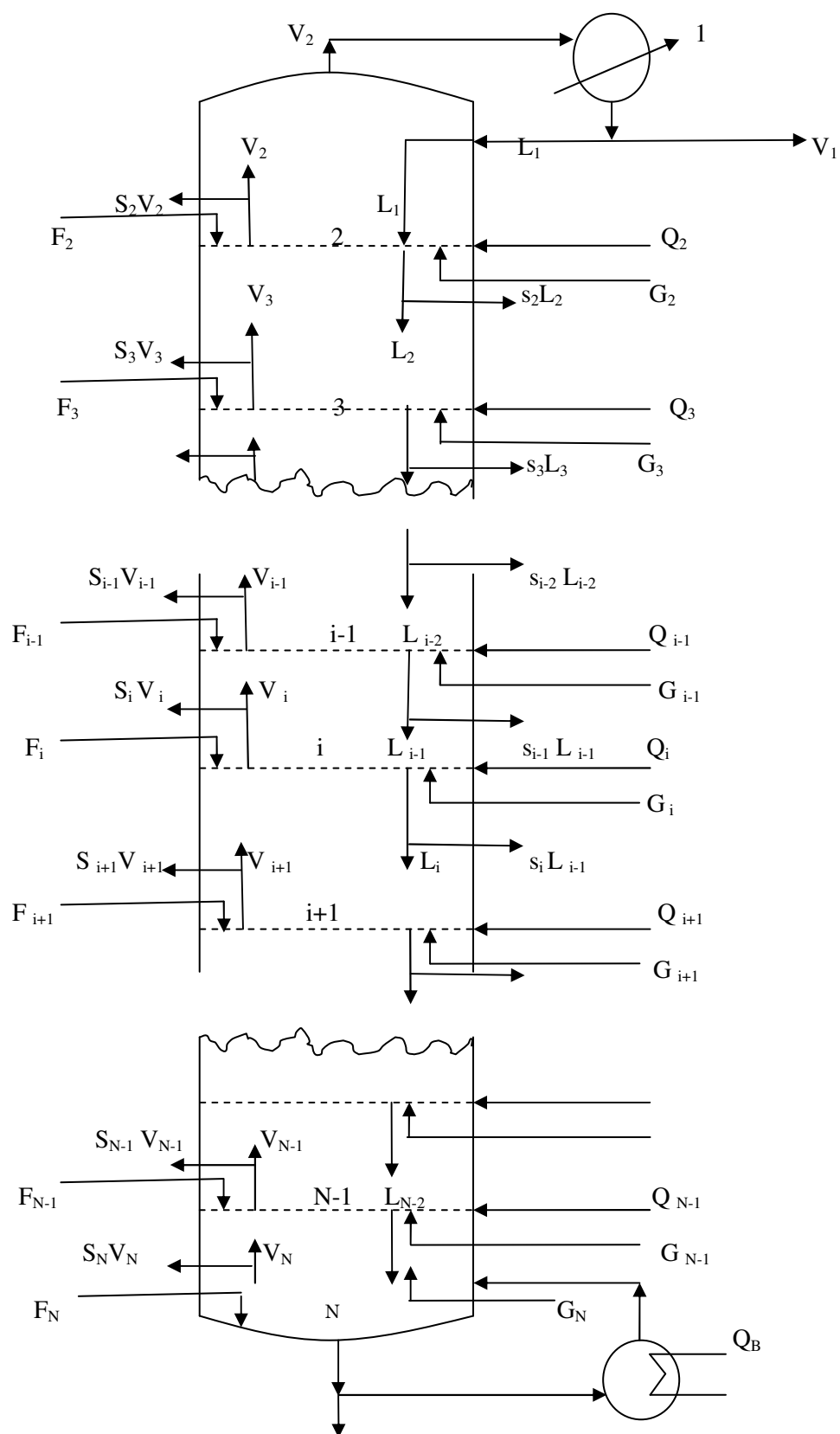


Figure-2.1 Configuration for reactive-distillation column

Consider a general, continuous, column consisting N-stage arranged in countercurrent cascade Separating C components. A schematic representation of Reactive Distillation is shown in figure 1, where stage 1 is a total condenser that produces a saturated liquid and stage N is a reboiler. Moreover, let chemical reactions occur at each stage in the liquid phase.

Assumptions used in model development:

1. Each stage is a perfectly mixed i.e. liquid composition at each stage is homogeneous and equal to the composition of liquid leaving the stage.
2. The vapor and liquid leaving any stage are in physical equilibrium.
3. Entrainment of liquid drops in vapor and occlusion of vapor bubbles in liquid are negligible.
4. Vapor molar holdup and vapor-phase chemical reactions were neglected.

The extent of liquid-phase reaction at each stage is governed by reversible kinetic rate expressions.

Fig-2.2 shows a schematic representation of a general reactive-distillation stage.

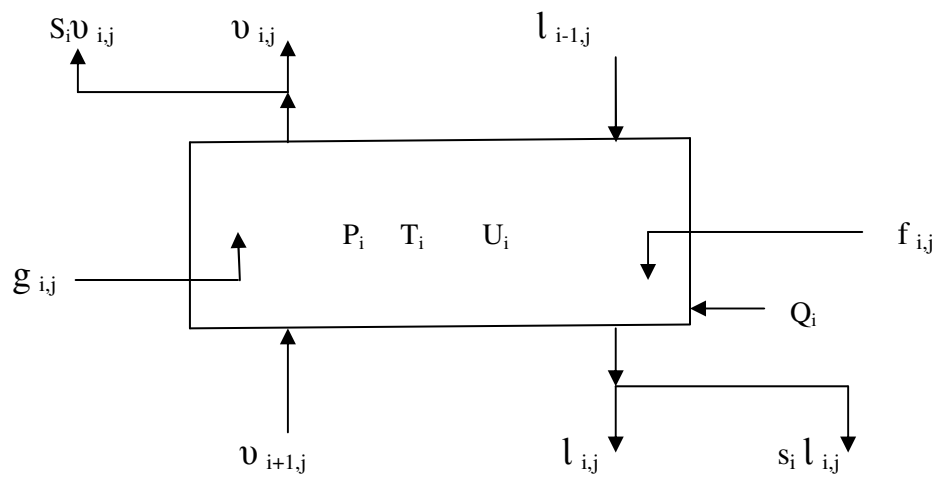


Figure-2.2 General reactive-distillation stage

The three types of functions which describe the physical and chemical processes on stage i are:

1. Component material balances:

$$M_{i,j} = (1+S_i) v_{i,j} + (1+s_i) l_{i,j} - v_{i+1,j} - l_{i-1,j} - f_{i,j} - g_{i,j} - U_i \sum_{n=1}^{NRX} v_{j,n} r_{i,n} \quad (2)$$

$j=1, \dots, C$
 $i=1, \dots, N$

Where the last term accounts for chemical reactions.

2. Energy balances:

$$E_i = (1+S_i)H_i + (1+s_i)h_i - H_{i+1} - h_{i-1} - h_{F,i} + H_{G,i} - Q_i \quad (3)$$

Unlike the component material balances, it is not necessary to take the heat of reaction into account because enthalpies are referred to the elements rather than to the components.

3. Equilibrium relationships:

$$Q_{i,j} = K_{i,j} l_{i,j} V_i / L_i - v_{i,j} \quad (4)$$

Where $Q_{i,j}$ is derived from the definition of $K_{i,j}$

$$K_{i,j} = y_{i,j}/x_{i,j} \text{ or } K_{i,j} x_{i,j} - y_{i,j} = 0 \quad (5)$$

Specifications for equations (2-4) include the total number of stages, stage locations of all feeds, side streams, and heat exchangers; all stage pressures and liquid-phase holdup volumes; and complete specification of each fresh feed. This leaves N specifications that must be made before solving the $N(2C + 1)$ equations for the corresponding $N(2C + 1)$ iteration variables. If the N specifications are N stage heat transfer rates, then the equations are solved for $2CN$ component molar flows and N stage temperatures. However, because condenser and reboiler duties are strongly dependent and can generally be specified independently over only narrow unknown ranges, other specifications are more desirable.

The total condenser stage is a special case which is not represented by equation (3). Let the component molar flows of the liquid distillate be called $v_{1,1}$, $v_{1,2}$, $v_{1,3}$, $v_{1,c}$. If the

reflux ratio is specified, the top-stage energy balance equation is changed to a reflux-ratio equation:

$$E_1 = L_1 - RV_1 \quad (6)$$

where R is the reflux ratio. The distillate composition, $v_{1,1}$, $v_{1,2}$, $v_{1,3}$, $v_{1,c}$ is the same composition as in the liquid leaving the condenser and, therefore, the equilibrium relations are not independent. the equilibrium equation for component 1 is replaced by the bubble point equation:

$$Q_{1,1} = \sum_{p=1}^c (K_{1,p} l_{1,p}) (V_1/L_1) - v_{1,p} \quad (7)$$

where component 1 must be present in the distillate in nontrivial amounts. Like the energy balance equation, the other equilibrium equations, $Q_{1,1}$ - $Q_{1,c}$, are substituted for by the component reflux equations; viz.

$$Q_{1,1} = l_{1,j} (V_1/L_1) - v_{1,j} \quad j=2, \dots, C \quad (8)$$

$r_{i,n}$ Chemical reaction rates in the liquid phase are modeled by reversible power-law kinetic rate equations. Vapor molar holdup and vapor-phase chemical reactions neglected. Because the material balance equations (1) are in the form out -minus- in, the term represents the moles of component j produced in the liquid phase. Therefore, $v_{i,j}$ the stoichiometric coefficient for component j in the n th chemical reaction, is positive if component j is created by the n th reaction (where the n subscript is omitted in following equation)

$$r_{i,n} = \sum_{p=1}^2 k_p \prod_{q=1}^{NRC} C_{i,q}^m = \sum_{p=1}^2 A_p \exp\left(-\frac{E_p}{RT_j}\right) \prod_{p=1}^{NRC} C_{i,q}^m \quad (9)$$

Where

$C_{i,q}$ = Concentration of component q on stage i

k_p = Reaction rate constant for the p th term, where $p=1$ indicates the forward reaction and $p=2$ indicates the reverse reaction; k_1 is positive and k_2 is negative.

m = Exponent of concentration

A_p = Pre exponential factor

E_p = Activation energy

Many attempts are taken to simulate model equations rigorously using different methods by putting different criteria for convergence. Since 1970, calculation procedures suitable for implementation on digital computers, for rigorously solving the mass, equilibrium, and energy balances in multicomponent, multistage reactive-distillation columns have been reported. The procedures, which involve the solution of large sets of combined nonlinear and linear equations, as well as applications, are summarized by Seader et al.,^[29]. From a numerical calculation procedure, these previously reported studies can be divided into three categories: (1) methods using tear variables (bubble-point method); (2) relaxation techniques (dynamic approach); and (3) methods incorporating the Newton-Raphson method.^[29]

The presence of a control system in a process introduces additional dynamics which, when coupled with dynamics of the unit, give rise to more complicity.

ASPEN technology uses inside-out simulator model in ASPEN PLUS, in computer program called RADFRAC and MULTIFRAC, these applications include^[1]:

1. Absorption, stripping, reboiled absorption, reboiled stripping, extractive distillation, and azeotropic distillation
2. Three phase(vapor-liquid-liquid) systems
3. reactive systems
4. highly nonideal system requiring activity-coefficient models
5. interlinked systems of separation units, including pumparounds, bypass, and external heat exchangers
6. narrow-boiling, wide-boiling, and dumbbell(mostly heavy and light components with little in between) feeds
7. presence of free water
8. wide variety of specification
9. use of murphree-stage efficiencies
10. RADFRAC is able to handle both equilibrium reactions as well as kinetically limited reactions

CHAPTER-3

STEADY STATE SIMULATION

Steady state simulation

3.1 CONFIGURATION-1(SINGLE COLUMN WITH SINGLE FEED):

The simulation of the ethyl acetate column is carried out using the rigorous distillation model RADFRAC from the flow sheet simulator Aspen Plus. In the simulations a property method WILSON is used from ASPEN global properties. The WILSON property method uses^[4]

- The Wilson activity coefficient model for the liquid phase
- The ideal gas equation of state for the vapor phase
- The Rackett model for liquid molar volume
- Henry's law for supercritical components

The WILSON model can describe strongly nonideal liquid solutions. The model cannot handle two liquid phases. In that case use other activity model like NRTL or UNIQUAC. The property methods with a vapor phase model that can be used up to moderate pressures, have the Poynting correction included in the liquid fugacity coefficient calculation. It can also handle any combination of polar and non-polar compounds, up to very strong non-ideality^[4].

The kinetic rate for the production of ethyl acetate in the liquid phase is adopted from the data of Holland^[27] for unanalyzed reaction^[5]. However we used different magnitude of rate constants to simulate the realistic catalyzed reaction which is about 100 times faster than unanalyzed one. Since the system is highly nonideal, highly non-ideal convergence is used in ASPEN PLUS. When RADFRAC encounters slow or difficult convergence for highly nonideal systems using the standard algorithm, highly non-ideal convergence is recommended^[4]. For any computer based simulation specification of problem is required and other variables are determined from specified variables. So the first simulation, Case 1, is made using the same specifications as Jin-Ho Lee, *et al.*,^[5]. The total number of stages is 13, including a total condenser and a partial reboiler; the numbering of the stages is top downward, column pressure is 1 atm. All the stages are reactive stages. The feed rate is 1.076 mol/s with a liquid distillate of 0.2080 mol/s, giving bottoms of 0.868mol/s. The feed is preheated to its bubble point at feed tray pressure and the reflux ratio is 10. The feed is fed to the sixth stage from the top and has the following mol-fraction composition:

acetic acid $x_1 = 0.4962$, ethanol $x_2 = 0.4808$, water $x_3 = 0.0229$ and ethyl acetate $x_4 = 0.0$. Holdup volumes are 1 liter and 10 liters, respectively, for the reboiler and each of the stages 1-12. Although these specifications are the same as those of Jin-Ho Lee, *et al.* the results of this study as discussed below are different. Comparisons with Ho Lee, *et al.* are not included here because they used considerably different vapor-liquid equilibrium ratios as compared to those used here. The simulation results obtained with ASPEN-PLUS are shown in Figures 3.2-3.3.

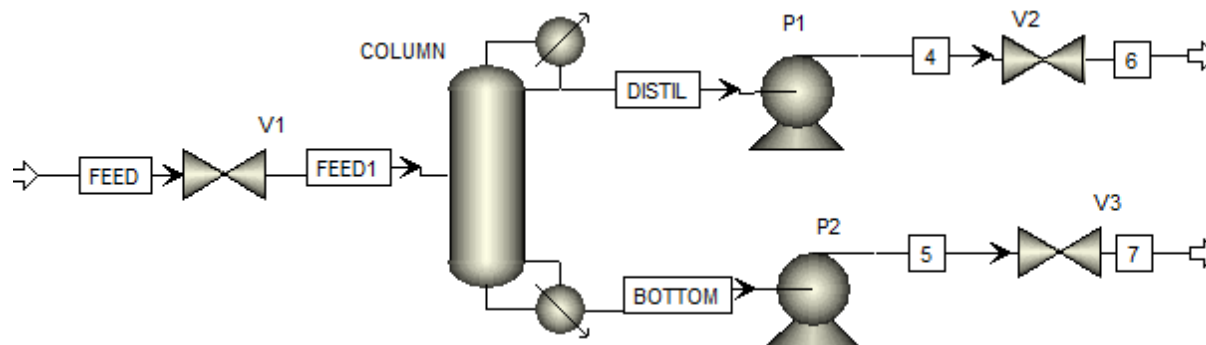


Figure-3.1 Single feed flow diagram in aspen plus user interface for RD

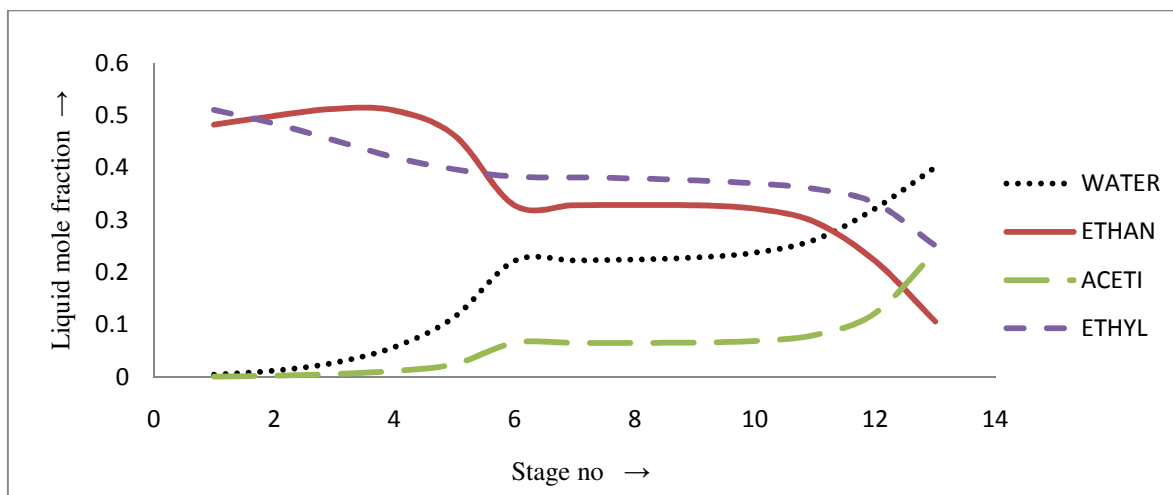


Figure-3.2 Stage wise composition profile

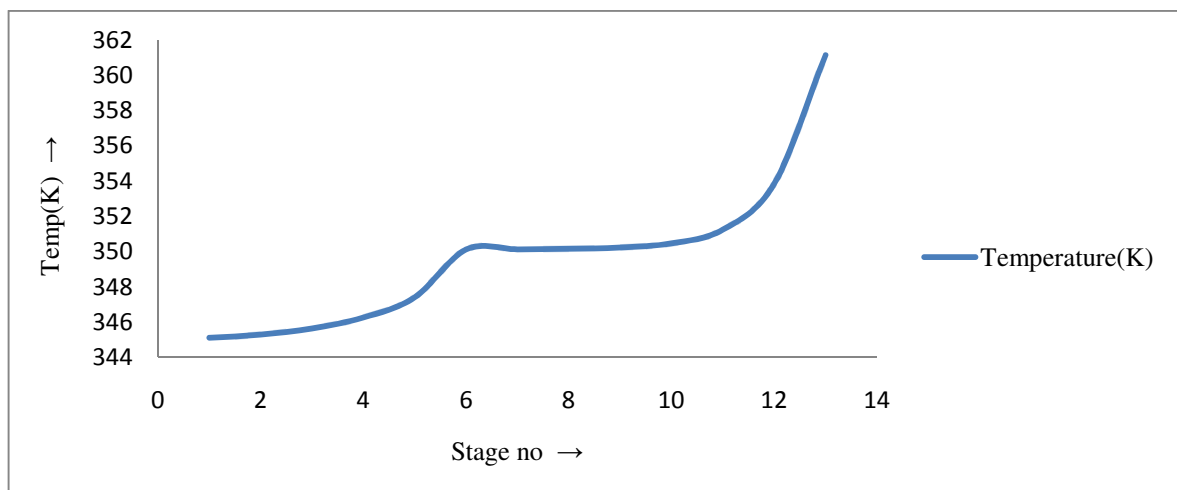


Figure-3.3 Stage wise temperature profile

Effect of reflux ratio on composition:

In steady state simulation of ethyl acetate reactive distillation column, keeping the entire specification constant as in configuration-1, reflux ratio is changed from 0.5 to 15. The liquid composition of ethyl acetate in distillate and in bottom is noted down from every simulation and a graph (fig 3.4) is plotted between reflux ratio and mole fraction of ethyl acetate. It is observed that after reflux ratio equal to 12 compositions is not changing significantly. So for further simulations reflux ratio equal to 12 is chosen.

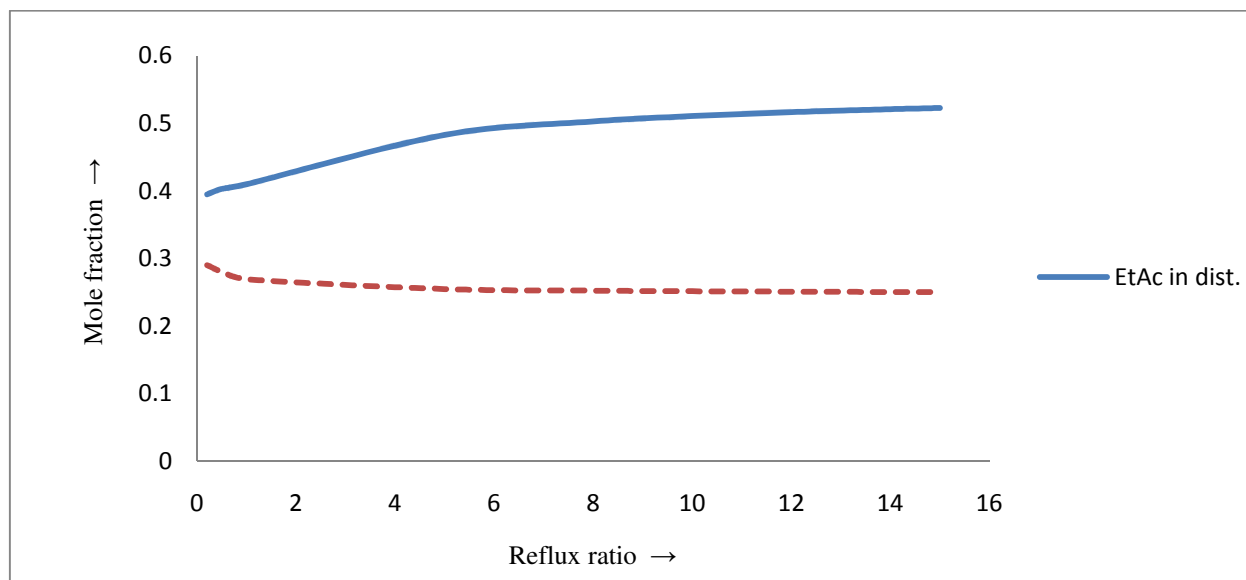


Figure-3.4 Effect of reflux ratio on liquid composition of ethyl acetate in distillate and bottom

Effect of distillate flow rate on composition:

Updating the value of reflux ratio from previous study result in steady state simulation of ethyl acetate reactive distillation column, remaining specification kept as it is and now distillate flow rate is varied and the liquid composition of ethyl acetate in distillate and in bottom is noted down from every simulation and a graph (fig 3.5) is plotted between distillate flow rate and mole fraction of ethyl acetate. The results of this study are listed in Table 1.

It is also observed that effect of distillate flow rate on composition throughout the column is much larger than the effect of reflux ratio.

Table 1(simulation result for changing distillate flow rate on composition):

| Distillate(mol/sec) | EtAc in distillate | EtAc in bottom |
|---------------------|--------------------|----------------|
| 0.1 | 0.5057 | 0.2928 |
| 0.15 | 0.5114 | 0.2752 |
| 0.2 | 0.5159 | 0.2546 |
| 0.208 | 0.5166 | 0.2510 |
| 0.25 | 0.5196 | 0.2307 |
| 0.3 | 0.5222 | 0.2027 |
| 0.35 | 0.5239 | 0.1697 |
| 0.4 | 0.5244 | 0.1306 |
| 0.45 | 0.5224 | 0.0835 |
| 0.5 | 0.5096 | 0.0271 |

From the graph it is observed that as distillate flow rate is changing liquid composition of ethyl acetate in distillate increases up to distillate flow rate equal to 0.4 mol/sec and then starts decreasing but concentration of ethyl acetate in bottom is decreasing continuously. Accordingly we choose distillate flow rate equal to 0.4 mol/sec future simulations.

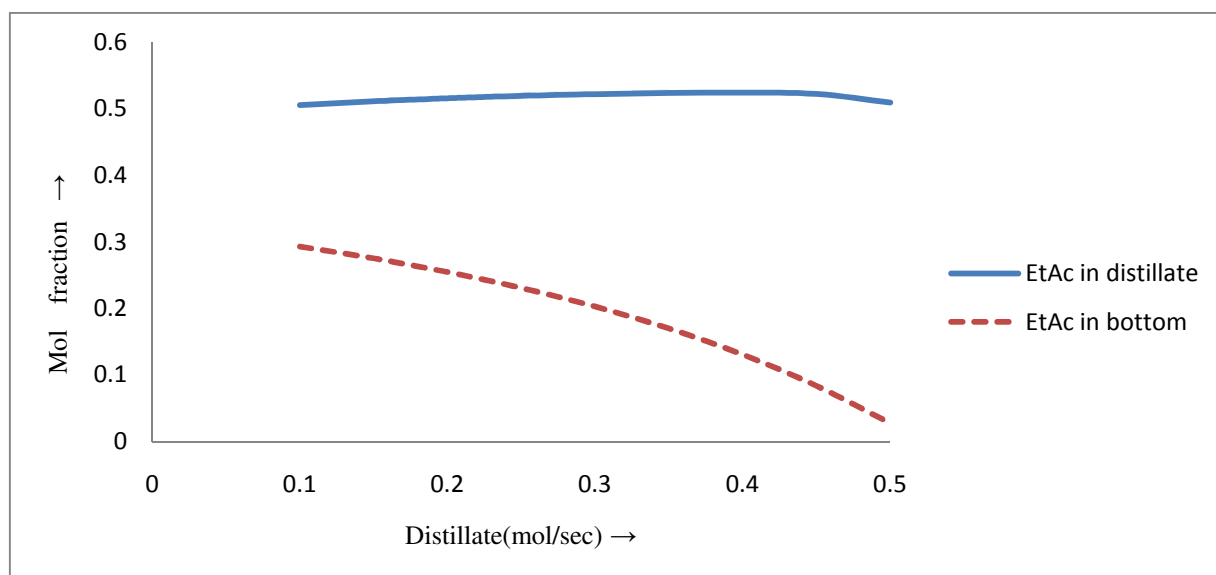


Figure-3.5 Effect of distillate flow rate on liquid composition of EtAc in distillate and bottom

Effect of total number stage in column on composition:

Considering pervious study results in steady state simulation of ethyl acetate reactive distillation column, our initial specification of reflux ratio and distillate flow rate has been changed to 12 and 0.4 respectively. Now changing the total number of stage in ASPEN PLUS data browser and the liquid composition of ethyl acetate in distillate and in bottom is noted down from every simulation. Fig. 3.6 shows the effect of number of equilibrium reactive stages in this column on liquid composition of ethyl acetate in distillate and bottom at a pressure of 1 atm. While total number of stage is varied it is assumed that feed plate is fixed ratio of total number of stage. The results of this study are listed in Table 2.

Table 2(simulation result for changing total no of stages in column on composition)

| Total number of stages | Feed stage | EtAc in Distillate | EtAc in bottom |
|------------------------|------------|--------------------|----------------|
| 8 | 3 | 0.4511 | 0.1525 |
| 10 | 4 | 0.4806 | 0.1408 |
| 13 | 6 | 0.5196 | 0.1313 |
| 16 | 7 | 0.5331 | 0.1291 |
| 20 | 9 | 0.5456 | 0.1274 |
| 22 | 9 | 0.5459 | 0.1421 |
| 23 | 10 | 0.5489 | 0.1535 |

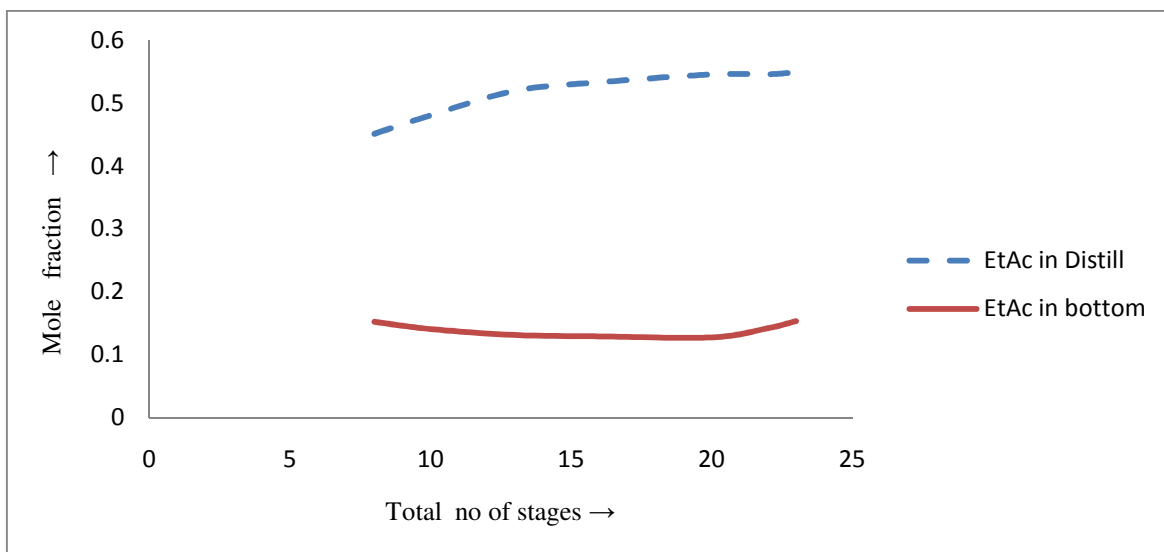


Figure-3.6 Effect of total no of stages in column on liquid composition of EtAc in distillate and bottom

From Table-2 and fig-3.6 it is observed that liquid concentration of ethyl acetate in distillate and in bottoms follow different trend. In distillate it increases but after total number stage equal to 20 increase is not significantly .while composition of ethyl acetate in distillate decreases up to total number of stages equal to 20 then starts increasing. So for further simulation total number of stage is equal to 20 and corresponding feed stage number is 9th.

Effect of different activity model on composition:

ASPEN PLUS has many in-built property models for liquid phase activity coefficient with combination of vapor phase model. In all Pervious study WILSON activity model is used. Now for this study activity model is changed and their effect on ethyl acetate concentration in distillate and bottom is studied. All other specifications are same as previous study; only total number of stage and feed stage in column is updated. Fig-3.7 shows composition profile of ethyl acetate in liquid phase throughout the column for different activity model. The results of this study are listed in Table 3.

Table 3(simulation results for different activity model on composition)

| Activity model | EtAc in Distillate | EtAc in bottom |
|----------------|--------------------|----------------|
| WILSON | 0.5457 | 0.1274 |
| NRTL | 0.4914 | 0.1715 |
| UNIFAC | 0.5217 | 0.1560 |
| UNIQUAC | 0.4903 | 0.1664 |

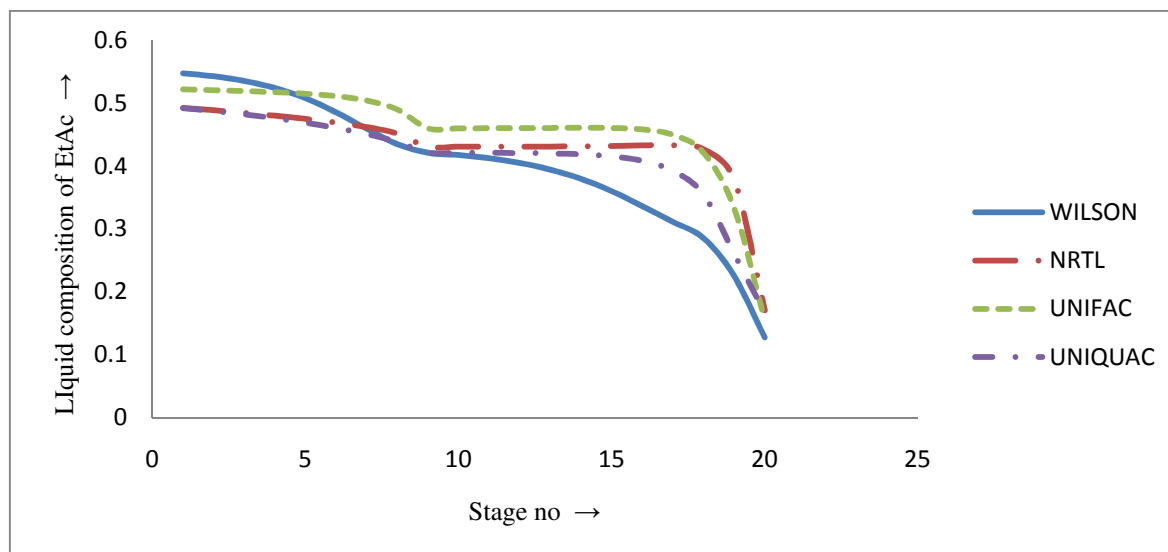


Figure-3.7 Composition profile of ethyl acetate in liquid phase for different activity model

Conclusion of configuration-1 study:

For a single-column, single-feed studies have been done. From results it can be easily seen that maximum concentration of liquid ethyl acetate in distillate is 0.55 and minimum concentration in bottom is 0.13. Our initial specification of reflux ratio, total number of stage, feed tray location and distillate flow rate has been changed but for all the sets of specifications studied, a nearly pure ethyl acetate distillate is not obtained and a high conversion is not possible.

3.2 CONFIGURATION-2(SINGLE COLUMN WITH DOUBLE FEED):

In this configuration of ethyl acetate reactive distillation simulation using ASPEN PLUS two separate saturated liquid feeds are used. Considering previous study results of configuration-1 specification is modified. In this configuration specifications are total number of stages is 20, including a total condenser and a partial reboiler; the numbering of the stages is top downward the column pressure is 1 atm. All the stages are reactive stages. Since column has two feed streams: a ethanol feed and a acetic acid feed, feed rate of ethanol is 0.5173 mol/sec and of acetic acid is 0.5339 mol/s with a liquid distillate of 0.40mol/s. The feed is preheated to its bubble point at feed tray pressure and the reflux ratio is 12. Holdup volumes are 1 liter and 10 liters, respectively, for the reboiler and each of the stages 1-19.

In the case of esterification, the order of volatility is ethyl acetate, ethanol, water and acetic acid. Liquid rich in acetic acid is fed to a stage between the top and the middle portion of the column. Vapor rich ethanol is fed to a stage between the middle portion and the bottom of the column. Reactant ethanol is absorbed into the liquid phase where the reaction takes place and product ethyl acetate is stripped from the liquid phase and carried out the top of the column. Thus, it is expected that the middle portion of the distillation tower is the chief reaction zone. The rectifying section fractionates the ethyl acetate out of the acetic acid, and the stripping section removes alcohol from water. Ideally, the ethyl acetate is the distillate and water is the bottoms product.

At first acetic acid is fed at stage 9th and ethanol at 10th. The ethanol feed stage location is varied in the simulations between stage 10 and stage 18 keeping acetic acid feed stage constant. A series of simulations are carried out with varying ethanol feed stage location. The ethanol feed is moved sequentially down the column from stage 10 to stage 18. A stage came where maximum purity of ethyl acetate in distillate and minimum in bottom is obtained; this stage number is chosen for ethanol feed location. Then acetic acid feed stage is varied keeping ethanol feed stage constant at new feed stage location. Acetic acid feed stage location varied between stage 9 and stage 2 and the liquid composition of ethyl acetate in distillate and in bottom is noted down. The results of this study are listed in Table 4.

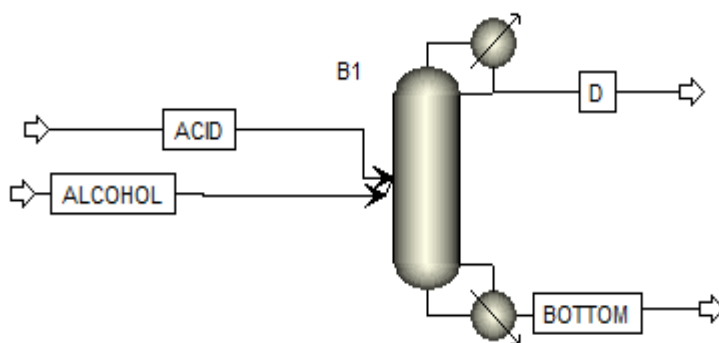


Figure-3.8 Double feed flow diagram in aspen plus user interface for Reactive Distillation

Table 4 (simulation result for double feed reactive distillation column)

| Acetic acid(stage no) | Ethyl alcohol(stage no) | Ethyl acetate (in dist.) | Ethyl acetate(in bottom) |
|-----------------------|-------------------------|--------------------------|--------------------------|
| 9 | 10 | 0.5480 | 0.1215 |
| 9 | 12 | 0.5505 | 0.1212 |
| 9 | 16 | 0.5528 | 0.1210 |
| 9 | 18 | 0.5533 | 0.1209 |
| 13 | 18 | 0.5534 | 0.1209 |
| 16 | 18 | 0.5536 | 0.1209 |
| 6 | 18 | 0.5557 | 0.1214 |
| 4 | 18 | 0.5641 | 0.1242 |
| 2 | 18 | 0.5841 | 0.1431 |

From results it is easily concluded that double feed in a single reactive distillation column gives more purity than single feed. However, the total amount of ethyl acetate in distillate just changes slightly.

3.3 Configuration-3(Double Reactive Distillation Column):

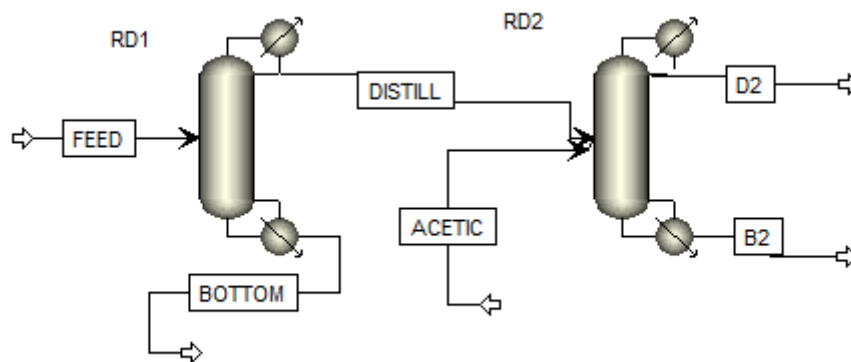


Figure -3.9 Double reactive distillation column diagram in aspen plus user interface

Our previous configuration study results show that in the distillate composition of ethanol is significant. So if a new reactive distillation column fed with acetic acid is added, ethanol might get converted to ethyl acetate.

For steady state simulation of ethyl acetate reactive distillation in ASPEN PLUS ,specification of both column is kept same as used in previous study i.e. the total number of stages is 20, including a total condenser and a partial reboiler; the numbering of the stages is top downward, column pressure is 1 atm. All the stages are reactive stages. The feed rate is 1.076 mol/s with a liquid distillate of 0.4 mol/s. The feed is preheated to its bubble point at feed tray pressure and the reflux ratio is 12. The feed is fed to the ninth stage from the top and has the following mol-fraction composition: acetic acid $x_1 = 0.4962$, ethanol $x_2 = 0.4808$, water $x_3 = 0.0229$ and ethyl acetate $x_4 = 0.0$. Holdup volumes are 1 liter and 10 liters, respectively, for the reboiler and each of the stages 1-12. Initially distillate from column-1 and additional acetic acid fed to reactive column-2 at same stage 9th. Quantity of acetic acid fed to reactive distillation column-2 is slightly more than ethanol which is present in distillate from column-1. Quantity of ethanol from reactive distillation column-1 can be found out by checking column profile then composition of liquid from stage-1. Hence quantity of acetic acid added to second reactive distillation column is 0.2 mol/sec. The simulation results obtained with ASPEN-PLUS are shown in Figure 3.10.

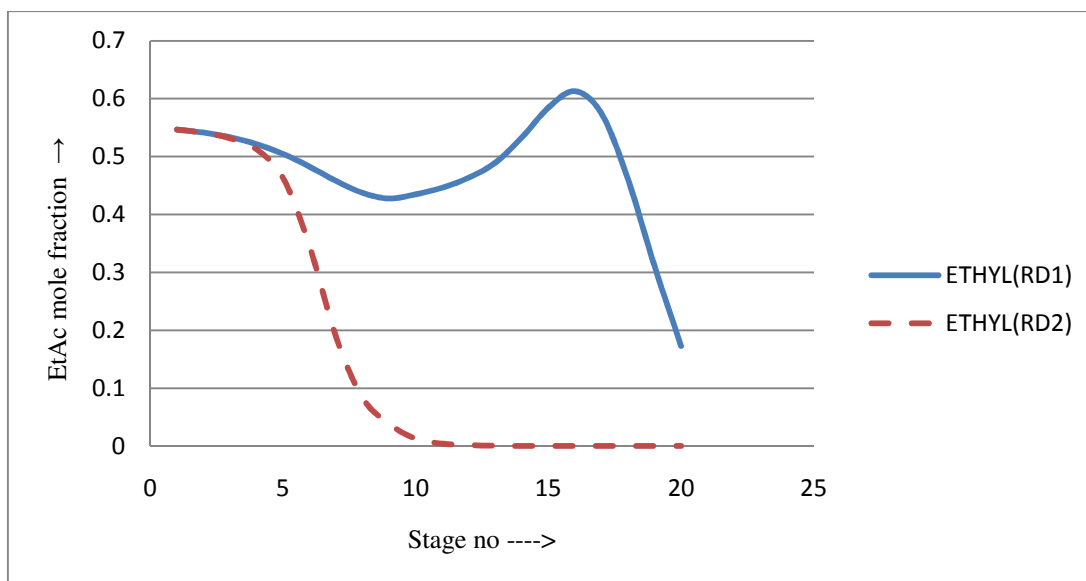


Figure-3.10 Stage-wise liquid composition profile of ethyl acetate in RD-1 and RD-2

Effect of changing feed stage to reactive column-2 on composition profile:

Now acetic acid is fed to a stage between the top and the middle portion of the reactive column (RD2), let's say on 4th stage and distillate from RD-1 is fed to a stage between the middle portion and the bottom of the column say 14th stage. Fig 11 shows stage-wise composition profile of ethyl acetate in liquid phase in both columns.

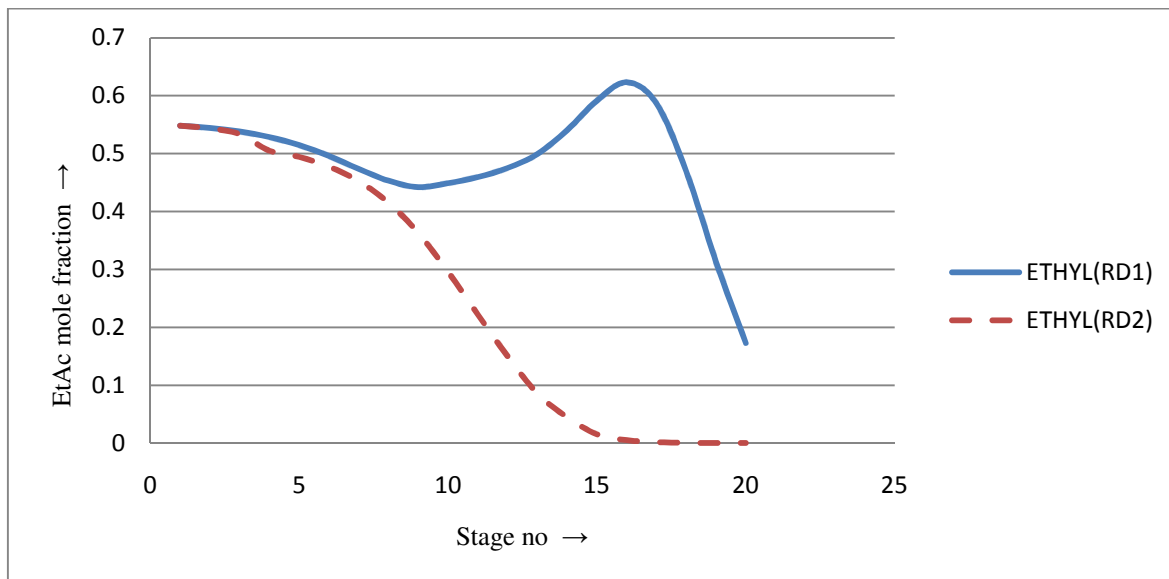


Figure- 3.11 Stage-wise liquid composition profile of ethyl acetate in RD-1 and RD-2 with different feed location

A similar simulation is also done with feeding acetic acid to a stage between the middle portion and the bottom of the reactive column-2 and distillate to a stage between the top and the middle portion of the reactive column-2.

Composition profile of reactive column-1 is not altered significantly on varying feed location to reactive column-1 and in reactive column-2 stage wise composition profile is altered but distillate and bottom composition of ethyl acetate remains almost constant.

3.4 CONFIGURATION-4(WITH RECYCLING OF HEATED SIDE STREAM/ PUMPAROUND):

Since esterification is an endothermic reaction, equilibrium shifts at higher temperatures toward the products; equilibrium conversion can be increased.

This fact leads to a new configuration with pumparounds to get more ethyl acetate in equilibrium limited esterification.

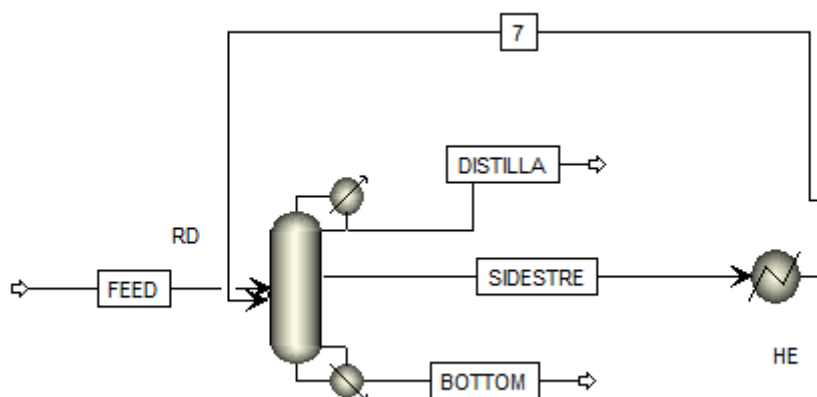


Figure-3.12 Reactive distillation column with heated side-stream in aspen plus user interface

To make pumparounds configuration for simulation of ethyl acetate reactive distillation column using ASPEN PLUS, Tear stream convergence or pumparounds form can be used. Both procedures are discussed below.

1. **Tear stream:** which stream is to be recycled initially break into its component stream and when specification of both stream is fairly close, the recycle loop is closed using *Tear* from data browser window of ASPEN PLUS e.g. in fig-3.12 initially stream-7 had two different stream; one is coming out from HE block (say 'output') and other is going to RD block (stream'7') with initial guess specification like temperature, pressure, flow rate and composition. Several runs were made in which guessed composition of '7' stream compared with simulation results of 'outlet' stream. When both are fairly closed, the loop 'output/7' is closed. The procedure for doing this involves three steps: (a) Delete either one of stream(say 'output') and (b) Reconnect another stream(click on the stream'7' and reconnect to block HE) (c) Go down near the bottom of list of items on the data browser window of ASPEN PLUS and click *convergence* and then *Tear*. Where the drop down menu is used to select stream'7'.

2. **Pumparounds form:** it is quite easier than Tear method and also takes comparatively less time in convergence. Pumparounds form is in-built with ASPEN PLUS. RadFrac can handle pumparounds from any stage to the same or any other stage. Use the Pumparounds form to enter all pumparound specifications. A pumparound can be either a partial or total drawoff of the: Stage liquid, First liquid phase, Second liquid phase or Vapor phase.

If the pumparound is a partial drawoff of the stage flow, we must enter two of the following specifications:

- Flow rate
- Temperature
- Temperature change
- Vapor fraction

Pressure specification is optional. The default pumparound pressure is the same as the source stage pressure. We can associate a heater or cooler with a pumparound. RadFrac assumes that the pumparound at the heater/cooler outlet has the same phase condition as the pumparound at the inlet. We can override the phase condition using the valid phases field on Pumparound Specifications sheet. RadFrac can return the pumparound to a stage using either the:

- On-stage option: RadFrac returns the pumparound to the specified stage. The pumparound is not flashed at the return stage pressure.
- Above-stage option: returns the pumparound to the column between two stages. The pumparound is flashed at the specified return stage pressure. RadFrac returns the liquid portion to the specified return stage, and the vapor portion to the stage above.

Effect of temperature difference across Heat Exchanger on composition of ethyl acetate:

In the first study of this configuration, temperature difference across heat exchanger is changed and effect on concentration of ethyl acetate in distillate and bottom is noted down for each simulation. pumaround is partial drawoff so two specification flow rate and temperature difference are specified. Draw stage is 9th and drawing phase is liquid and return is “on-stage” type to 8th stage. The results of this study are listed in Table 5.

Table-5(study result of varying ΔT across Heat Exchanger on composition)

| ΔT across Heat Exchanger | Ethyl acetate(in Distillate) | Ethylacetate(in bottom) |
|----------------------------------|------------------------------|-------------------------|
| 5 | 0.5436 | 0.1277 |
| 10 | 0.5434 | 0.1277 |
| 15 | 0.5431 | 0.1277 |
| 20 | 0.5430 | 0.1277 |
| 25 | 0.5428 | 0.1278 |
| 30 | 0.5425 | 0.1278 |

From study results it is observed that ethyl acetate purity decreases slightly in distillate and in bottom remains almost constant.

Effect of draw stage location and return stage location:

Keeping draw stage at 9th as in previous study, pumaround return stage is varied and result is summarized in table below. At a particular combination of draw stage and return stage temperature difference across heat exchanger is also varied.

Table 6a (study result of pumparound return location on composition)

| Pumparound | ΔT across Heat Exchanger | Ethyl acetate(in Distillate) | Ethyl acetate(in bottom) |
|---------------|----------------------------------|------------------------------|--------------------------|
| Inlet stage-8 | 5 | 0.5436 | 0.1277 |
| Draw stage-9 | 15 | 0.5431 | 0.1277 |
| | 25 | 0.5428 | 0.1278 |
| Inlet stage-6 | 5 | 0.5333 | 0.1292 |
| Draw stage-9 | 15 | 0.5325 | 0.1293 |
| | 25 | 0.5315 | 0.1295 |

Now pumparound inlet stage location is moved downward in lower section of column.

Table 6b (study result of pumparound return location on composition)

| pumparound | ΔT across Heat Exchanger | Ethyl acetate(in Distillate) | Ethyl acetate(in bottom) |
|----------------|----------------------------------|------------------------------|--------------------------|
| Inlet stage-10 | 5 | 0.5456 | 0.1275 |
| Draw stage-9 | 15 | 0.5454 | 0.1275 |
| | 25 | 0.5425 | 0.1275 |
| Inlet stage-12 | 5 | 0.5454 | 0.1275 |
| Draw stage-9 | 15 | 0.5453 | 0.1275 |
| | 25 | 0.5451 | 0.1275 |

From table it is observed that ethyl acetate increases when inlet of pumparound is between middle and bottom section of column.

CHAPTER-04

DYNAMIC SIMULATION

ASPEN PLUS steady state simulation files are indispensable for dynamic simulation. So first steady state simulation is done and these files are exported to ASPEN DYNAMICS. In preparation for exporting the steady-state flow sheet into Aspen Dynamics, all equipment is needed to be sized. Column diameters are calculated by Aspen tray sizing. Reflux drums and column bases are sized to provide 5 min of holdup when 50% full, based on the total liquid entering the surge capacity. Pumps and control valves are not necessary for steady-state simulation, but they are vital for a realistic dynamic simulation. Providing sufficient pressure drop over a control valve at design conditions with the valve at some fraction opening (typically 50%) is crucial for dynamic controllability. These are specified to give adequate dynamic rangeability. Typical valve pressure drops are 2 atm.^[3]

Steady state simulation of ethyl acetate reactive distillation column configuration-1(single column, single feed) is exported to ASPEN DYNAMICS. The diameter of a distillation column is determined by the maximum vapor velocity. If this velocity is exceeded, the column liquid and vapor hydraulics will fail and the column will flood. Reliable correlations are available to determine this maximum vapor velocity. Since the vapor flow rates change from tray to tray in a nonequimolar overflow system, the tray with the highest vapor velocity will set the minimum column diameter. Aspen Plus has an easy-to-use tray sizing capability. Click the sub-item Tray Sizing under the COLUMN block, and then click *New* and *OK* for the identification number, where the column sections to be sized and the type of tray can be entered.

Aspen Dynamics calculates tray pressure drops rigorously, and they change with vapor and liquid rates. In steady state simulation constant pressure is assumed throughout the column but before exporting files to aspen dynamic a pressure drop equal to 0.006 atm is chosen.^[3]

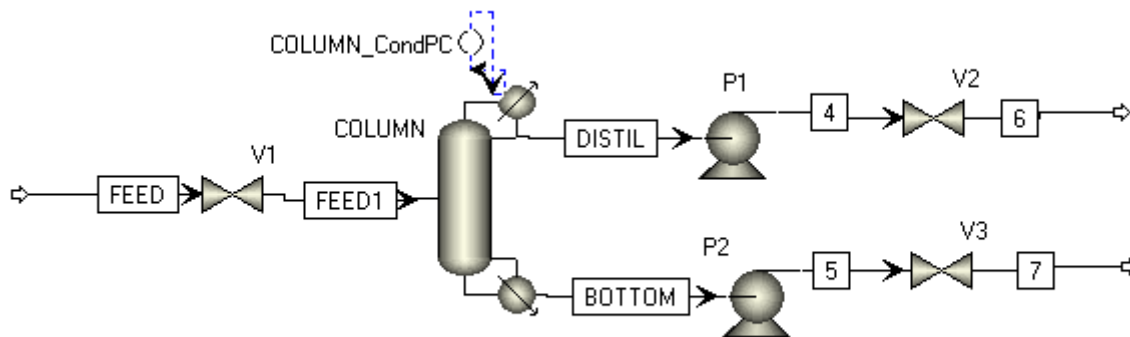


Figure-4.1 Initial screen in ASPEN DYNAMICS for single column with single feed

Plate hydraulics is taken from Jin-Ho Lee et al ^[5] and values are entered by clicking the dynamics button on the top toolbar. The window that opens has several page tabs. On the Reflux Drum page tab, the appropriate diameter and length are entered. The same is done on the Sump page tab. Finally, the Hydraulics page tab is clicked, and on which stage numbers (2 through 19) and the column diameter (0.6 m) are entered. The values of weir height and tray spacing are 0.05 and 0.340 m, respectively.

The initial flow sheet has some default controllers already installed (Fig-4.1). In this single-column process there is only one default controller, the pressure controller. It is configured to measure condenser pressure and manipulate condenser heat removal. At a minimum, four additional controllers must be added to achieve effective operation of the column:

1. Reflux drum-level controller
2. Base-level controller
3. Feed-flow controller
4. Tray temperature controller

For liquid level control our control objective is to keep each liquid level within a certain range around a desired set point. Consequently, proportional control alone is satisfactory. For flow control systems a PI controller is satisfactory because it eliminates offset and remains acceptable speed of response. For temperature control a PID controller would be the most appropriate, because it can allow high gains for faster response without undermining the stability of system^[28]

4.1 Installing level controller (LC1 & LC2)

For level controller-1(LC1) process variable (PV) is liquid level on the last stage of the column (stage 20) and manipulated variable (OP) flow rate through valve V3. The action of the controller should be *Direct* because if the level increases, the signal to the valve should increase (PV↑, OP↑) to remove more bottoms. In some columns, base level is controlled by manipulating a valve in the feed to the column. In that control structure, the base level controller action should be *Reverse*. Since we want proportional-only control, the controller gain is set equal to 2 and the integral time is set at a very large number (9999 min). The second-level controller LC2 for the reflux drum is installed and connected in the same way. The PV signal comes from the level on stage 1. The OP

goes to valve V2. A direct-acting proportional-only controller is specified. The default value of the controller output range is 0–100%

4.2 Installing flow controller

The next basic controller that we need to set up is a flow controller on the feed. A PID controller is placed on the flow sheet. Its PV signal is the molar flow-rate of feed stream *FEED*. Its OP signal goes to valve V1. After opening the Tuning page tab and clicking the Initialize Values button, we set the controller to be Reverse-acting, and use conventional flow controller tuning ($K_C=0.5$ and integral time= 0.3 min) ^[3]. The most common error in setting up the flow controller is to forget to specify *Reverse* action. Since flow control is very fast and essentially algebraic, it seems to help the numerical integrator to use some filtering in a flow controller. The Filtering page tab is selected, the Enable filtering box is checked, and a small filter time constant (0.1 min) is typed.

4.3 Installing temperature controller

A PIDIncr controller is installed on the flow sheet in the normal way. The important difference between PID and the PIDIncr controller is that PIDIncr has a built-in relay–feedback test capability, which make this dynamic test a breeze. A temperature controller is used to maintain a tray temperature in the column. Looking at the temperature profile in Aspen Plus, we see that stage 19 displays a fairly steep slope. Its temperature is 361.70K. The PV is selected to be the temperature on stage 19. The OP is selected to be the reboiler heat input QRebR. The controller action should be set at *Reverse* because if the tray temperature is going up, the reboiler heat input should be decreased. The program is run to make sure that everything works okay without a lag or a dead time in the loop. Now we back up and insert a dead time element on the flow sheet between the column and the TC temperature controller. The reason for installing the controller initially without the dead time element is to avoid initialization problems that sometimes crop up if we attempt to install the dead time and the controller all in one shot.

RELAY FEEDBACK TEST:

Applications of relay feedback to distillation columns, reactor/separator recycle plants, fuel processors, and reactive distillation columns have been reported in the literature, and the method is shown to be effective for nonlinear processes. The relay feedback test proposed by Åström and Hägglund^[31] has received much attention in the process control community. Luyben^[32] was among the first to use the relay feedback test for system identification and it is shown to be effective for

highly nonlinear chemical processes, namely, high-purity distillation columns. Chang *et al.* [33] derived the transfer function from the relay feedback test with increased accuracy. The autotune variation (ATV) identification method has become a standard practice in chemical process control.

In ASPEN DYNAMICS we specify a Closed loop ATV as the Test method. The default value of the Relay output amplitude is 5%, which is usually good. For a very nonlinear column, the amplitude may have to be reduced. After several (4–6) cycles have occurred, test is finished and the Tyreus–Luyben Tuning rule is selected. The resulting controller settings are gain $K_C = 4.84$ and integral time is 8.43 min.

4.4 Installing composition controller

Composition measurement typically has larger dead time and lags than does temperature Control. We assume a 3-min dead time in the composition measurement. First, we add a PIDIncr controller to the flow sheet and make the appropriate connections and do not use a dead time, which will be added later. The controller should be set to *Reverse*. The PV is the mole fraction of ethyl acetate in the distillate stream. The OP is reboiler heat input. A composition transmitter range of 0–0.05 mF ethyl acetate is used, after the simulation is run, a 3-min dead time is inserted. Initialization and Dynamic runs are made to converge to steady-state conditions. Then a relay-feedback test is run. Controller is updated with Tyreus–Luyben Tuning.

4.6 Cascade Control Structure:

Temperature control has the advantage of being fast, but it may not hold the product purity constant. Composition control is slow, but it will drive product purity to the desired value. The final control structure studied is a cascade combination of composition and temperature control that achieves both fast control and the maintenance of product purity.

The tray temperature controller is the secondary controller. It is set up in exactly the same way as we did in the previous section. It looks at tray temperature and manipulates reboiler heat input. However, its set point is not fixed. The set point signal is the output signal of the composition controller, which is the primary controller.

The tuning of the secondary temperature controller remains unchanged. The primary composition controller must be retuned since its output signal is now a temperature set point.

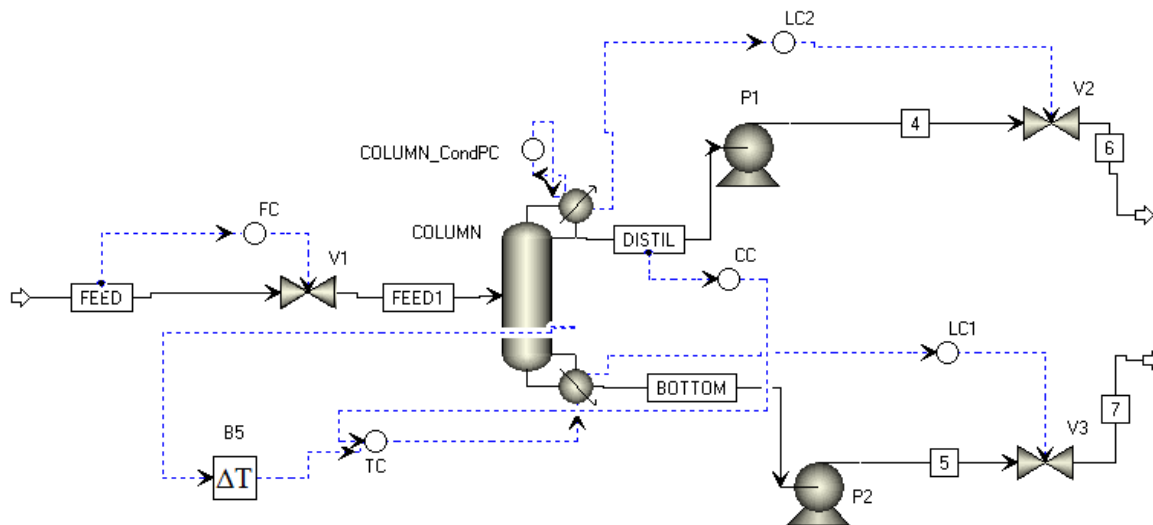


Figure-4.2 Configuration-1 with cascade control structure in ASPEN DYNAMICS

4.7 Performance Evaluation of Control Structure:

We want to see how well control structures developed above perform in the face of disturbances, specifically, how close to the desired values of temperature and composition these variables are maintained, both at steady state and dynamically. A disturbance in feed flow and in composition is made and the transient responses are plotted.

At time equal 1 h, the set point of the feed flow controller is changed from 1.073 to 1.4mol/sec.

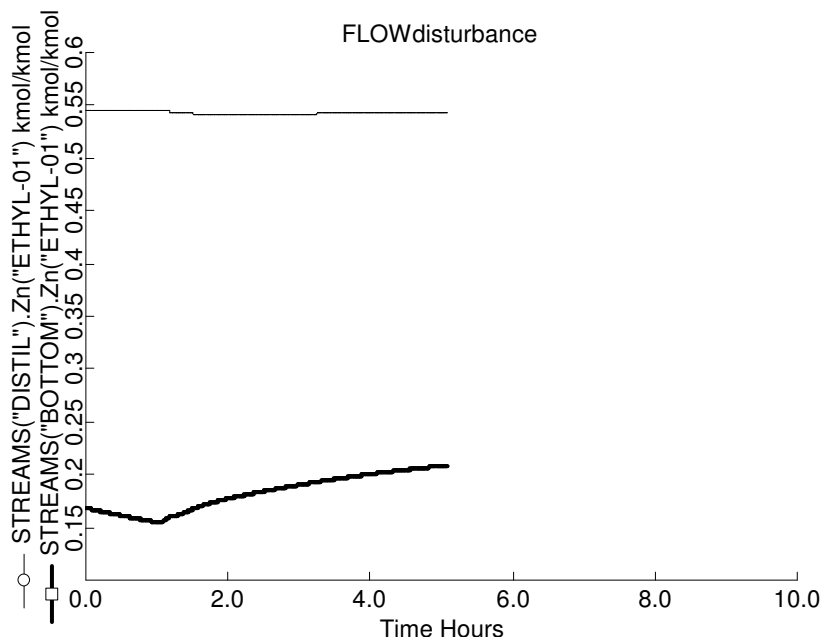


Figure-4.3 Dynamic response of composition in bottom and distillate subjected to feed flow disturbance

Ethyl acetate in Bottom composition attains a new steady state while no change in distillate composition.

Now mole fractions of ethanol and acetic acid are changed from 0.48/0.50 to 0.52/0.46.

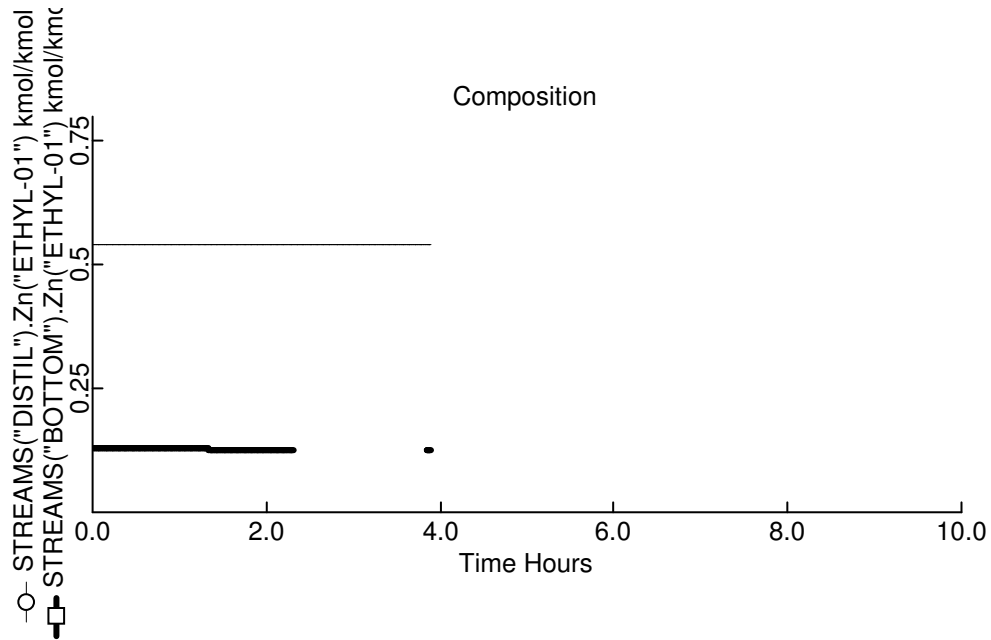


Figure-4.4 Dynamic response of composition in bottom and distillate subjected to feed composition disturbance

Set point change in composition controller(0.45 to 0.55)

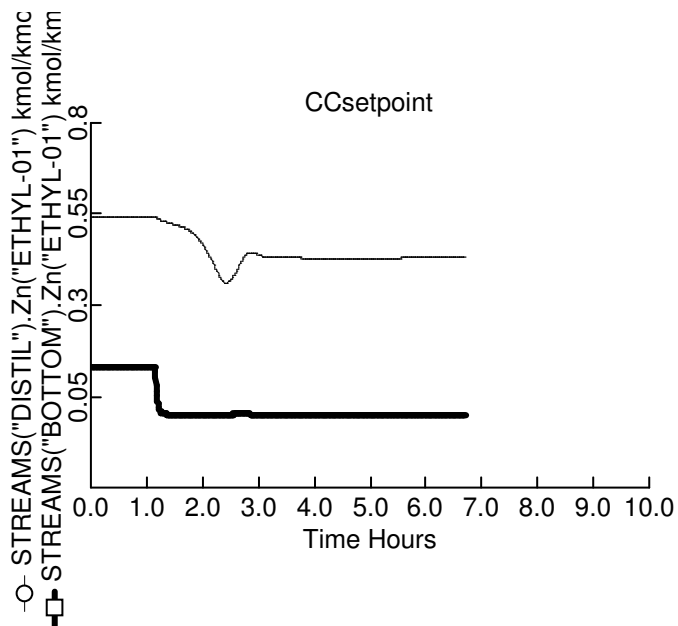


Figure-4.5 Dynamic response of composition in bottom and distillate subjected to distillate set-point disturbance

Conclusion:

In this work, all the results are obtained from steady-state and dynamic simulations using ASPEN PLUS 2006.2 and ASPEN DYNAMICS 2006.2 programs for ethyl acetate reactive distillation column. Double feed gives more purity of ethyl acetate than single feed. Double column configuration with additional acetic acid fed to second column drastically reduces ethyl acetate in bottom but in top changes slightly. Providing pumparound in middle and bottom section of column increases ethyl acetate in distillate. Dynamic study demonstrates the value of dynamic simulation in the analysis of alternative control schemes for reactive distillation process.

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